PROJECT NUMBER: 1752
PROJECT TITLE: Molecular Structure Determination
PROJECT LEADER: G. Vilcins
PERIOD COVERED: November, 1989

I. CARBON 13 NMR ANALYSIS OF NICOTINE ON CLAY

A. Objective: To determine the degree of loading of nicotine in the clay and the type of chemical species present.

B. Results: A nicotine saturated clay was extracted to remove the nicotine. The residual nicotine in the clay was detected by ^{13}C CPMAS NMR. The signals were broadened by paramagnetic iron atoms in the clay structure. By measuring the signal intensity as a function of the cross polarization contact time, the ^{1}H rotating frame relaxation rates were measured. The relaxation appeared to be non-exponential either because of the suppression of proton spin diffusion due to the paramagnetic centers or because there are two or more nicotine sites in the clay with intrinsically different relaxation rates. By assuming two nicotine sites with different rotating frame relaxation rates, a reasonable approximation of the relaxation curves were obtained. The relative proportion of the two types of nicotine in the extracted clay differed from a saturated clay sample but the relaxation rates appeared to be the same.

C. Conclusions: Nicotine adsorbed in clay may be distributed between two different sites. Extraction of the nicotine caused a redistribution of the residual nicotine between the two sites. This suggested a stronger affinity for one site over the other.

D. References:

Wooten, J., "^{13}C CPMAS NMR of Nicotine Adsorbed on Clay," memo to J. Seeman, November 1, 1989.

II. WATER COLUMN STUDIES FOR THE ART PROCESS

A. Objective: The effect of monopotassium citrate in the aqueous extraction of carbon dioxide tobacco solubles was determined.

B. Results: The SFE/SFC instrument was configured to perform online extraction and gas chromatographic separation. Nicotine extracted in the carbon dioxide was monitored by ultraviolet detection at 274 nm. The effluent was passed through a restrictor into a heated split injector where it was cold trapped on the front end of a capillary column. Chromatograms were obtained of each tobacco extraction performed with no water, water, and 6% monopotassium citrate. Peaks were identified by GC/FT-IR/MS.

C. Conclusions: Final evaluation of the data is in progress.

D. References:

Shafer, K., PM Notebook #8789 pp. 55.

2022192896

Source: https://www.industrydocuments.ucsf.edu/docs/hyjm0000

PROJECT NUMBER: 1757
PROJECT TITLE: Analytical Flavor Specifications
PROJECT LEADER: M. L. Zimmermann
PERIOD COVERED: November, 1989

ANALYTICAL FLAVOR SPECIFICATIONS

A. **Objective**: To develop analytical and sensory specifications for incoming flavors and materials for use at the Flavor Center and other QA facilities. To provide analytical certification on export flavors manufactured at the Flavor Center for FRG compliance.

B. **Results**:

Contaminant analyses continue on tobacco flavors, revisions of processed oils, aroma chemicals and possible vendor alternates for specific materials. All codes processed by the Flavor Center have now been completed. Approximately 30 remaining materials will be obtained from the specific usage sites for contaminant analysis.

Certification for the PMI samples continues and two specific analyses are no longer required.

A modified head space sampling procedure was evaluated for use in the certification process by the Dionex equipment. Various vial/septa combinations, sample matrix changes and heating times were examined. Alternatives to these procedures are being investigated.

Samples received from K. Podraza and H. Secor were evaluated for nicotinic acid content. Additional components were examined by a variety of GC, LC and GC/MS techniques.

Round robin participation for Flavor Center QA was conducted to assist in the sugar analysis procedure refinement.

Training of QA specialist on the FRG methodology and basic instrument operation, spectral interpretation, GC and HPLC methodology continues.

Two members participated in a special smoke panel group.

C. **Plans**: Continue the certification of the PMI samples, begin the certification of all flavors currently on hand at the Flavor Center and continue the specification work for the second vendor.

2022192897

Source: https://www.industrydocuments.ucsf.edu/docs/hyjm0000